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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the mimetic diagram showing the gestalt of operation of the manufacturing installation of the organic electroluminescence devices of this invention.

[Drawing 2] It is the typical sectional view showing one example of the organic electroluminescence-devices body manufactured by this invention.

[Drawing 3] It is the typical sectional view showing other examples of the organic electroluminescence-devices body manufactured by this invention.

[Drawing 4] It is the typical sectional view showing another example of the organic electroluminescence-devices body manufactured by this invention.

[Drawing 5] It is the typical sectional view showing the example of the organic electroluminescence devices manufactured by this invention.

[Drawing 6] It is the graph which shows the drive luminescence time dependency of the relative luminescence brightness to the initial luminescence brightness of the organic electroluminescence devices in an example 1 and the examples 1 and 2 of a comparison.

[Description of Notations]

- 1 Substrate
- 2 Anode Plate
- 3 Organic Luminous Layer
- 3a Electron hole transportation layer
- 3b Electronic transportation layer
- 3c Hole injection layer
- 4 Cathode
- 5 Protective Layer
- 6 Sealing Compound
- 7 Tooth-Back Glass
- 10, 10A, 10B Organic electroluminescence-devices body
- 21 27 Dry box
- 22, 23, 24, 25, 26 Working-level month vacuum chamber
- 22a, 23a, 24a, 25a, 26a Vacuum chamber for conveyance
- 22b, 23b, 24b, 25b, 26b Robot arm
- 22c, 23c, 24c, 25c, 26c, 27c Gate valve
- 22d, 23d, 24d, 25d, 26d Gate valve

[Translation done.]

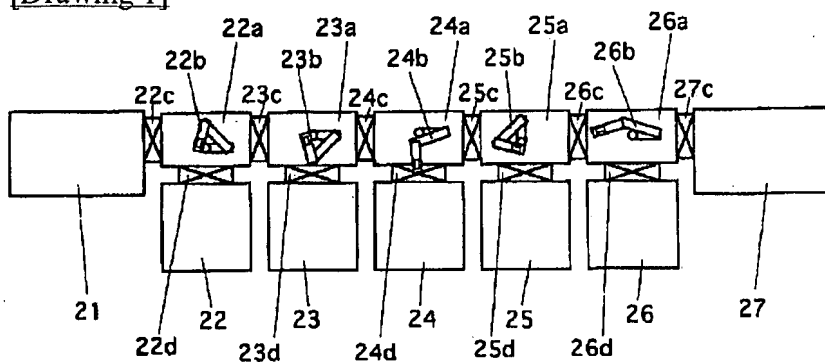
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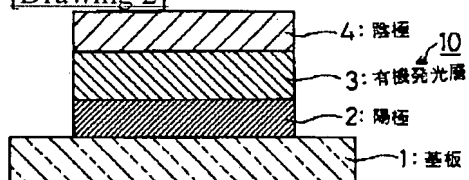
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DRAWINGS

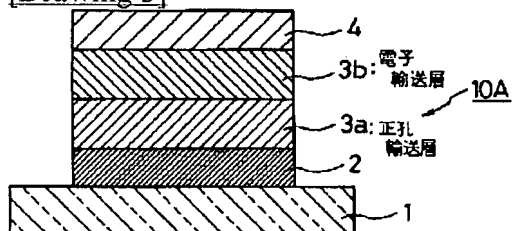
[Drawing 1]



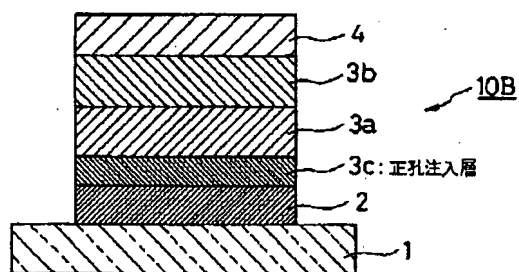
[Drawing 2]



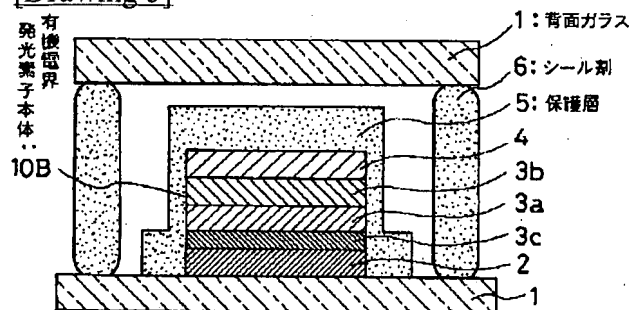
[Drawing 3]



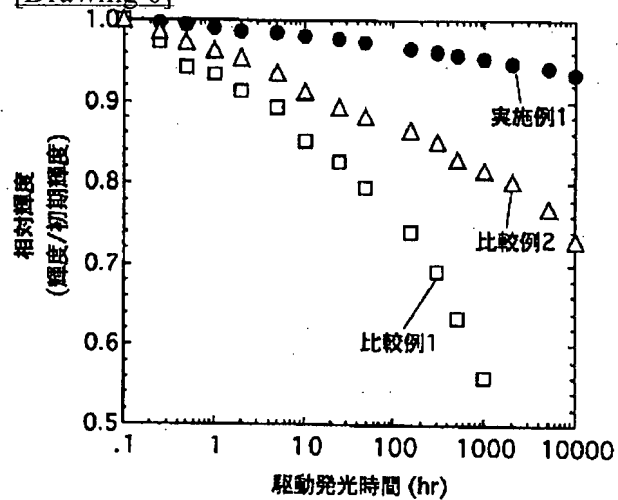
[Drawing 4]



[Drawing 5]



[Drawing 6]



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CLAIMS

[Claim(s)]

[Claim 1] In the equipment for manufacturing the organic electroluminescence devices which come to prepare two or more stratified deposits on a substrate The vacuum chamber for conveyance with carrying-in opening and taking-out opening of a substrate, and the working-level month vacuum chamber which stands in a row through the bulb which can pass [that vacuum transfer cutoff is possible and] said substrate in this vacuum chamber for conveyance, A migration means to be installed in this vacuum chamber for conveyance, and to transport a substrate into this working-level month vacuum chamber from said carrying-in opening, and to transport to said taking-out opening from this working-level month vacuum chamber, It is the organic electroluminescence-devices manufacturing installation equipped with the 1st thru/or the n-th unit for migration and processing which has this processing means for stratified deposit formation established in this working-level month vacuum chamber. So that the substrate introduced in this manufacturing installation may be transported from carrying-in opening of the 1st unit to the n-th unit through this unit The manufacturing installation of the organic electroluminescence devices characterized by connecting taking-out opening of the unit of the substrate migration direction upstream to carrying-in opening of the unit of the substrate migration direction downstream.

[Claim 2] The manufacturing installation of the organic electroluminescence devices which are equipment for manufacturing the organic electroluminescence devices which come to prepare the seal member surrounding this stratified deposit while preparing two or more stratified deposits on a substrate, and are characterized by to have the facility for preparing said seal member, without exposing to atmospheric air after forming all stratified deposits in the manufacturing installation of the organic electroluminescence devices which prepared the following stratified deposit, without exposing to atmospheric air after preparing one stratified deposit.

[Claim 3] The manufacturing installation of the organic electroluminescence devices characterized by the equipment for preparing said stratified deposit on a substrate being a manufacturing installation according to claim 1 in claim 2.

[Claim 4] The manufacture approach of the organic electroluminescence devices characterized by to prepare said seal member, without exposing to atmospheric air after forming in the approach of manufacturing the organic electroluminescence devices which come to have the stratified deposit by which deposition formation was carried out, and which has an anode plate, an organic luminous layer, and cathode at least, and a seal member surrounding this stratified deposit to the shape of a layer on a substrate and this substrate, without exposing an organic luminous layer and cathode to atmospheric air within a vacuum tub at least among these stratified deposits.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention is the equipment and the approach of manufacturing the thin film mold light emitting device which emits light, applying electric field to the luminous layer which consists of an organic compound in detail about the manufacturing installation and its manufacture approach of organic electroluminescence devices, and relates to the equipment and the approach of mass-production nature being good and manufacturing efficiently organic electroluminescence devices excellent in the luminescence life property by low cost.

[0002]

[Description of the Prior Art] Organic electroluminescence devices have the descriptions, such as spontaneous light, a thin shape, and a high angle of visibility, and attract attention as the new source of flat-surface mold luminescence thru/or a display device.

[0003] In order to manufacture organic electroluminescence devices conventionally After forming transperence electric conduction film, such as ITO (indium stannic-acid ghost), by approaches, such as a spatter, on a glass substrate, After carrying out pattern processing, forming a lower electrode, installing this substrate in a vacuum deposition tub and forming an organic luminous layer etc. by approaches, such as heating vacuum evaporatio, It once took out from this vacuum tub, and the metal layer, the protective layer, etc. were formed by another vacuum tub, it took out from this vacuum tub after that, and sealing of the component section was performed.

[0004] By the above-mentioned conventional approach, in order to form multilayer structure, such as an organic electron hole transportation layer, an organic luminous layer, cathode, and a protective layer, the vacuum tub was returned to ordinary pressure from the vacuum each time, the raw material needed to be replaced and masks needed to be exchanged. Moreover, even when two or more vacuum tubs were prepared, the substrate between vacuum tubs needed to be changed. For this reason, since a substrate was exposed to atmospheric air between previous vacuum evaporatio and the next vacuum evaporatio and a vacuum evaporatio layer was exposed to oxygen and the moisture in atmospheric air, there was a problem that the property of a component fell. Moreover, in order to repeat the vacuum suction of a vacuum tub, and ordinary pressure release, production time was long and the trouble of being bad also had productive efficiency.

[0005] The approach of delivering a substrate and carrying out sequential vacuum evaporatio, having arranged two or more vacuum deposition tubs, and maintaining a vacua at the surroundings of it for the meantime focusing on one vacuum conveyance tub, for the purpose of performing the vacuum deposition process from after pattern processing of the transperence electric conduction film to formation of a protective layer consistently as an approach of solving these troubles, is tried (JP,8-111285,A).

[0006]

[Problem(s) to be Solved by the Invention] However, by this approach, from the description on the structure where of a conveyance tub will grow large especially if; substrate size which has a limitation in

the number of the vacuum evaporationno tubs which can be arranged around a conveyance tub becomes large, since; substrate which does not turn to a mass-production machine will be discharged outside from a vacuum tub in front of a seal process after the protection stratification and will be exposed to atmospheric air, it had troubles, such as; with the difficult reinforcement of a component.

[0007] this invention person solves the above-mentioned conventional trouble, and it aims at offering the equipment and the approach of mass-production nature being good and manufacturing the organic electroluminescence devices which were excellent in the property by low cost.

[0008]

[Means for Solving the Problem] In equipment for the manufacturing installation of the organic electroluminescence devices of claim 1 to manufacture the organic electroluminescence devices which come to prepare two or more stratified deposits on a substrate The vacuum chamber for conveyance with carrying-in opening and taking-out opening of a substrate, and the working-level month vacuum chamber which stands in a row through the bulb which can pass [that vacuum transfer cutoff is possible and] said substrate in this vacuum chamber for conveyance, A migration means to be installed in this vacuum chamber for conveyance, and to transport a substrate into this working-level month vacuum chamber from said carrying-in opening, and to transport to said taking-out opening from this working-level month vacuum chamber, It is the organic electroluminescence-devices manufacturing installation equipped with the 1st thru/or the n-th unit for migration and processing which has this processing means for stratified deposit formation established in this working-level month vacuum chamber. It is characterized by connecting taking-out opening of the unit of the substrate migration direction upstream to carrying-in opening of the unit of the substrate migration direction downstream so that the substrate introduced in this manufacturing installation may be transported from carrying-in opening of the 1st unit to the n-th unit through this unit.

[0009] If it is the manufacturing installation of these organic electroluminescence devices, the vacuum chamber for conveyance of the 1st unit, The working-level month vacuum chamber of the 1st unit, the vacuum chamber for conveyance of the 1st unit, the vacuum chamber for conveyance of the 2nd unit, The working-level month vacuum chamber of the 2nd unit, and the vacuum chamber for conveyance of the 2nd unit, by carrying out sequential migration of the substrate in the order of the vacuum chamber for conveyance of the unit of ** (n-1), the vacuum chamber for conveyance of the n-th unit, the working-level month vacuum chamber of the n-th unit, and the vacuum chamber for conveyance of the n-th unit Without exposing a substrate to atmospheric air, a vacua can be maintained and a stratified deposit can be formed efficiently (in addition). It is also possible to form a stratified deposit, without going via the working-level month vacuum chamber of the unit of arbitration, or it is also possible to return from the 3rd unit to the 2nd unit, and to form a stratified deposit.

[0010] And since the unit this migration and for processing is connected by the vacuum chamber for conveyance, the number of arbitration can be connected and the number of working-level month vacuum chambers for forming a stratified deposit is not restricted. It can respond also to the increment in the stratified deposit which extension of this unit is also easy a deposit and vapor-deposits it easily, and a manufacturing cost can be held down. Moreover, even when substrate size is large, the vacuum chamber for conveyance does not grow large.

[0011] The manufacturing installation of the organic electroluminescence devices of claim 2 is equipment for manufacturing the organic electroluminescence devices which come to prepare the seal member which surrounds this stratified deposit while preparing two or more stratified deposits on a substrate. After forming all stratified deposits in the manufacturing installation of the organic electroluminescence devices which prepared the following stratified deposit, without exposing to atmospheric air after preparing one stratified deposit, it is characterized by having the facility for preparing said seal member, without exposing to atmospheric air.

[0012] If it is the manufacturing installation of these organic electroluminescence devices, after forming a stratified deposit in a substrate, the seal member surrounding this stratified deposit can be prepared without exposing a substrate to atmospheric air, and organic electroluminescence devices excellent in the luminescence life property can be manufactured.

[0013] As for the equipment for preparing a stratified deposit on a substrate, in this equipment, it is desirable that it is said manufacturing installation according to claim 1.

[0014] The stratified deposit with which deposition formation of the manufacture approach of the organic electroluminescence devices of claim 4 was carried out on the substrate and this substrate at the shape of a layer and which has an anode plate, an organic luminous layer, and cathode at least, In the approach of manufacturing the organic electroluminescence devices which come to have a seal member surrounding this stratified deposit, after forming without exposing an organic luminous layer and cathode to atmospheric air within a vacuum tub at least among these stratified deposits, it is characterized by preparing said seal member, without exposing to atmospheric air.

[0015] By this approach, since it carries out without exposing a substrate to atmospheric air consistently from formation of a stratified deposit to formation of a seal member, the organic electroluminescence devices which were remarkably excellent in the luminescence property and the luminescence life property can be manufactured.

[0016]

[Embodiment of the Invention] With reference to a drawing, the gestalt of operation of this invention is explained below at a detail.

[0017] First, the configuration of the organic electroluminescence devices manufactured by this invention with reference to drawing 2 -5 is explained.

[0018] Drawing 2 -4 are the typical sectional view showing the example of structure of the organic electroluminescence-devices body of the organic electroluminescence devices manufactured by this invention, and drawing 5 is the typical sectional view showing the organic electroluminescence devices which come to carry out sealing of such an organic electroluminescence-devices body by the seal member.

[0019] drawing 2 -5 -- setting -- 1 -- a substrate and 2 -- an anode plate and 3 -- an organic luminous layer and 3a -- an electron hole transportation layer and 3b -- for cathode and 5, a protective layer and 6 are [an electronic transportation layer and 3c / a hole injection layer and 4 / tooth-back glass, and 10, 10A and 10B of a sealing compound and 7] organic electroluminescence-devices bodies.

[0020] A substrate 1 serves as a base material of organic electroluminescence devices, and to excel in properties, such as optical property, thermal-resistance, surface precision, mechanical-strength, lightweight nature, and gas barrier property, is demanded. As a substrate 1, generally, although the plate of a quartz or glass, a metal plate, a metallic foil and plastic film, a sheet, etc. are used, transparent synthetic-resin substrates, such as a glass plate, and polyester, the poly meta acrylic rate, a polycarbonate, the poly ape phone, are suitable.

[0021] The anode plate 2 formed on the substrate 1 plays the role of the hole injection to the organic luminous layer 3. As for this anode plate 2, conductive polymers, such as halogenation metals, such as metallic oxides, such as oxide of metals, such as aluminum, gold, silver, platinum, nickel, palladium, and platinum, an indium, and/or tin, and copper iodide, carbon black or Pori (3-methylthiophene), polypyrrole, and the poly aniline, etc. are usually preferably formed of an indium stannic-acid ghost (ITO). Formation of an anode plate 2 is usually performed by the sputtering method, a vacuum deposition method, etc. in many cases. When using particles, such as metal particles, such as silver, and copper iodide, carbon black, a conductive metallic-oxide particle, conductive polymer impalpable powder, etc., this can be distributed in a suitable binder resin solution, and an anode plate 2 can also be formed by applying on a substrate 1. Moreover, when using a conductive polymer, an anode plate 2 can also be formed by forming a thin film on the direct substrate 1 by electrolytic polymerization, or applying a conductive polymer on a substrate 1. The laminating of the different matter is carried out and an anode plate 2 can also form it.

[0022] The thickness of an anode plate 2 changes with existence of a demand of transparency. When transparency is needed, it is desirable to usually make the permeability of the light into 80% or more preferably 60% or more, and 5-1000nm of thickness is usually about 10-500nm preferably in this case. When opaque [an anode plate 2] and good, you may be the same ingredient as a substrate 1. Moreover, it is also possible to carry out the laminating of the different electrical conducting material on an anode

plate 2.

[0023] The organic luminous layer 3 formed on an anode plate 2 is formed in inter-electrode [to which electric field were given] from the ingredient which conveys efficiently the electron poured in from the electron hole and cathode 4 which were poured in from the anode plate 2, and is made to recombine it, and emits light efficiently by recombination. Usually, as this organic luminous layer 3 is shown in drawing 3 for improvement in luminous efficiency, making it the functional discrete type divided into electron hole transportation layer 3a and electronic transportation layer 3b is performed.

[0024] In functional discrete-type organic electroluminescence-devices 10A shown in drawing 3 , as an ingredient of electron hole transportation layer 3a, the hole-injection effectiveness from an anode plate 2 is high, and it is required to be the ingredient which can convey the poured-in electron hole efficiently. For that purpose, ionization potential is small, moreover, hole mobility is large, it excels in stability further, and it is required that it should be hard to generate the impurity which serves as a trap at the time of manufacture and use.

[0025] The aromatic series diamine compound which connected the 3rd class aromatic amine units, such as a 1 and 1-bis(4-G p-tolylamino phenyl) cyclohexane, as such an electron hole transportation ingredient, for example, 4 and 4'-screw [N-(1-naphthyl)-N-phenylamino] Aromatic amine which two or more fused aromatic rings permuted by the nitrogen atom including two or more tertiary amine represented with a biphenyl, The aromatic series triamine which has starburst structure with the derivative of triphenyl benzene, Aromatic series diamines, such as N, N'-diphenyl-N, the N'-bis(3-methylphenyl) biphenyl -4, and 4'-diamine, alpha, alpha, alpha', alpha'-tetramethyl - alpha, alpha'-bis(4-G p-tolylamino phenyl)-para xylene, The compound which the aromatic series diamino radical permuted by the triphenylamine derivative unsymmetrical in three dimensions and the BIRENIRU radical as the whole molecule, The aromatic series diamine which connected the 3rd class aromatic amine unit by ethylene, The aromatic series diamine which has styryl structure, the thing which connected the aromatic series tertiary amine unit by the thiophene radical, Starburst mold aromatic series triamine, a benzyl phenyl compound, the thing that connected tertiary amine by the fluorene radical, A triamine compound, a bis-pyridylamino biphenyl, N and N, N-triphenylamine derivative, The aromatic series diamine which has phenoxazine structure, a diamino phenyl phenanthridine derivative, a hydrazone compound, a silazane compound, a silanamine derivative, a phosphamine derivative, the Quinacridone compound, etc. are mentioned. These compounds may be used independently, and two or more sorts may be mixed and used if needed.

[0026] In addition, polymeric materials, such as a polyvinyl carbazole, polysilane, poly FOSUFAZEN, a polyamide, a polyvinyl triphenylamine, a giant molecule that has a triphenylamine frame, a giant molecule which connected the triphenylamine unit by the methylene group etc., and polymethacrylate containing aromatic amine, can be used as an ingredient of electron hole transportation layer 3a in addition to the above-mentioned compound.

[0027] Laminating formation of the electron hole transportation layer 3a is carried out on said anode plate 2 by forming these electron hole transportation ingredients with vacuum evaporation technique, the sputtering method, electron beam vacuum deposition, etc.

[0028] In forming electron hole transportation layer 3a with a vacuum deposition method, after paying electron hole transportation ingredients to the crucible installed in the vacuum housing and exhausting the inside of a vacuum housing to about 10 - 4Pa with a suitable vacuum pump, a crucible is heated, an electron hole transportation ingredient is evaporated, and electron hole transportation layer 3a is formed on the anode plate 2 on the substrate 1 which carried out opposite arrangement at the crucible.

[0029] Thus, when forming electron hole transportation layer 3a, a low-battery drive can be enabled by doping the metal complex of aromatic carboxylic acid and/or a metal salt, a benzophenone derivative and a thio benzophenone derivative, and fullerene by 10-3 - 10% of the weight of concentration, and making the electron hole as a free carrier generate as an acceptor further.

[0030] 10-300nm of thickness of electron hole transportation layer 3a is usually 30-100nm preferably. In order to form uniformly the thin electron hole transportation layer of such thickness, generally a vacuum deposition method is used well.

[0031] Moreover, as shown in drawing 4, forming hole injection layer 3c between electron hole transportation layer 3a and an anode plate 2 is also performed, in order to raise hole-injection effectiveness further and to improve the adhesion force to the anode plate 2 of the whole organic layer. As an ingredient used for hole injection layer 3c, ionization potential is low, conductivity is high, the ingredient which can form a stable thin film thermally on an anode plate 2 further is desirable, and a phthalocyanine compound and a porphyrin compound are used. By making such hole injection layer 3c intervene, the effectiveness that the power surge when carrying out the continuation drive of the component by constant current is also controlled is acquired at the same time the driver voltage of an early component falls. It is possible to raise conductivity with hole injection layer 3c as well as electron hole transportation layer 3a doping an acceptor.

[0032] 2-100nm of thickness of hole injection layer 3c is usually 5-50nm preferably. In order to form the thin hole injection layer of such thickness uniformly, generally a vacuum deposition method is used well.

[0033] Electronic transportation layer 3b formed on electron hole transportation layer 3a consists of compounds which can convey the electron from cathode in the direction of electron hole transportation layer 3a efficiently in inter-electrode [to which electric field were given].

[0034] As an electronic transportability compound used for electronic transportation layer 3b, the electron injection effectiveness from cathode 4 is high, and it is required to be the compound which can convey the poured-in electron efficiently. For that purpose, an electron affinity is large, moreover electron mobility is large, it excels in stability further, and to be the compound which the impurity which serves as a trap at the time of manufacture and use cannot generate easily is demanded.

[0035] As an ingredient which fulfills such conditions, metal complexes, such as aromatic compounds, such as a tetra-phenyl butadiene, and an aluminum complex of 8-hydroxyquinoline, a cyclopentadiene derivative, a peri non derivative, an OKISA diazole derivative, a bis-styryl benzene derivative, a perylene derivative, a coumarin compound, a rare earth complex, a JISUCHIRIRU pyrazine derivative, p-phenylene compound, a thiadiazolo pyridine derivative, a pyrrolo pyridine derivative, a NAFUCHI lysine derivative, etc. are mentioned.

[0036] Generally electronic transportation layer 3b using these compounds can play in coincidence the role which conveys an electron, and an electron hole and the role which brings about luminescence in the case of electronic recombination.

[0037] When electron hole transportation layer 3a has a luminescence function, electronic transportation layer 3b may play only the role which conveys an electron.

[0038] although being the purpose which changes the luminescent color, for example, doping fluorochromes for laser, such as a coumarin, by using the aluminum complex of 8-hydroxyquinoline as a host ingredient etc. is performed while raising the luminous efficiency of a component -- this invention -- also setting -- the above-mentioned organic electronic transportability ingredient -- a host ingredient -- carrying out -- various kinds of fluorochromes -- 10-3-10-mol % -- the luminescence property of a component can be further raised by doping.

[0039] 10-200nm of thickness of electronic transportation layer 3b is usually 30-100nm preferably.

[0040] Although electronic transportation layer 3b can also be formed by the same approach as electron hole transportation layer 3a, a vacuum deposition method is usually used.

[0041] In addition, as an organic luminous layer 3 of a monolayer mold which does not perform functional separation as shown in drawing 2, it is Pori (p-phenylenevinylene) and Pori which were mentioned previously. [2-methoxy-5-(2-ethylhexyloxy)-1 and 4-phenylenevinylene] Polymeric materials, such as Pori (3-alkyl thiophene), the system which mixed luminescent material and an electronic transition ingredient to macromolecules, such as a polyvinyl carbazole, are mentioned.

[0042] Cathode 4 plays the role which pours an electron into the organic luminous layer 3. Although the ingredient used as cathode 4 can use the ingredient used for said anode plate 2, in order to perform electron injection efficiently, its low metal of a work function is desirable, and suitable metals or those alloys, such as tin, magnesium, an indium, calcium, aluminum, and silver, are suitable for it. The thickness of cathode 4 is usually comparable as an anode plate 2.

[0043] When a work function carries out the laminating of the stable metal layer to atmospheric air further highly on this cathode, the stability of a component can be increased in order to protect the cathode which consists of a low work function metal. Metals, such as aluminum, silver, nickel, chromium, gold, and platinum, are used for the metal layer for this purpose.

[0044] In addition, drawing 2 -4 can show an example of the component body adopted by this invention, and this invention can be applied to the component body of lamination as shown below in addition to the thing of illustration.

[0045] An anode plate / electron hole transportation layer / electronic transportation layer / volume phase / cathode, an electronic transportation layer / cathode besides anode plate / electron hole transportation layer / electronic transportation layer/, By an electronic transportation layer / the cathode above-mentioned lamination besides electronic transportation layer / volume phase / cathode, anode plate / hole injection layer / electron hole transportation layer / electronic transportation layer / volume phase / cathode, anode plate / hole injection layer / electron hole transportation layer / electronic transportation layer/[besides anode plate / electron hole transportation layer / electronic transportation layer/] A volume phase is for raising contact to cathode and an organic layer. An aromatic series diamine compound, The layer which consisted of the Quinacridone compound, a naphthalene derivative, the organic silicon compound, an organic phosphorous compound, a compound that has N-phenyl carbazole frame, an N-vinylcarbazole polymer, etc. can be illustrated. 2-100nm of thickness of a volume phase is usually 5-30nm preferably. Instead of preparing a volume phase, the field which contains the ingredient of the above-mentioned volume phase 50% of the weight or more near the cathode interface of an organic luminous layer and an electronic transportation layer may be prepared.

[0046] Moreover, in order that other electronic transportation layers may raise the luminous efficiency of organic electroluminescence devices further, it is required for the compound which laminating formation is further carried out on an electronic transportation layer, and is used for this electronic transportation layer that the electron injection from cathode should be easy and electronic transport capacity should be still larger. As such an electronic transportability ingredient, the system which distributed an OKISA diazole derivative and them to resin, such as a polymethyl methacrylate (PMMA), a phenanthroline derivative or n mold hydrogenation amorphous carbonization silicon, n mold zinc sulfide, n mold zinc selenide, etc. are mentioned. 5-200nm of thickness of other electronic transportation layers is usually 10-100nm preferably.

[0047] In order to raise the stability and dependability of such organic electroluminescence devices, it is necessary to carry out the seal of the whole stratified deposit, such as the whole component, i.e., the anode plate formed on the substrate, an organic luminous layer, and cathode.

[0048] Below, sealing concerning this invention is explained with reference to drawing 5 . In addition, although the case where sealing of the protective layer 5 is prepared and carried out on the cathode 4 of organic electroluminescence-devices body 10B of the structure shown in drawing 4 is illustrated to drawing 5 , it cannot be overemphasized that you may be drawing 2 , the bodies 10 and 10A of organic electroluminescence devices shown in 3, or the organic electroluminescence-devices body of other lamination.

[0049] in drawing 5 , a protective layer 5 is formed in order to protect the cathode 4 and the organic layer under it -- having -- **** -- SiOx, and GeO, MgO and Ta 2O5 etc. -- an oxide and SiNx etc. -- sulfides, such as a nitride and GeS, and MgF2 etc. -- a fluoride etc. is used. As the formation approach, the vacuum evaporation technique by resistance heating, a spatter, electron beam vacuum deposition, etc. are used. Among these, although many vacuum evaporation technique is used in order to control the damage at the time of the protection stratification, using together the spatter formed into the low damage, and vacuum evaporation technique and a spatter is also performed. Generally thickness of a protective layer 5 is set to 100nm - 10 micrometers, and is suitably determined according to membranous precision, stress, and other properties and military requirements.

[0050] After forming a protective layer 5, except for the electrode section pulled out to particular parts, such as the circumference of a substrate 1, sealing is performed in order to intercept the component section (stratified deposit) from the external world. The seal member consists of a sealing compound

(encapsulant) 6 and tooth-back glass 7 at least, after it applies a sealing compound 6 to a substrate 1 or tooth-back glass 7, it contacts both, it hardens a sealing compound 6, and completes sealing.

[0051] In this invention, it takes all over a dry box, without exposing the substrate 1 after forming a protective layer 5 to atmospheric air in this sealing, and this sealing is performed.

[0052] The thing of the space where the moisture content (water vapor content) occupied in environmental gas compared with a dry box here in usual atmospheric air was controlled low can be said, and inert gas, such as air, nitrogen, and an argon, and other handling matter and gas which does not react can be used as environmental gas. As an approach of making it into a dry environment, the gas dried [argon], for example permutes the inside of a box, and there is the approach of maintaining a dry environment by continuing passing small quantity [every] gas also after that. With this approach, it is [about / dew-point]. -10 degrees C (about 1600 ppm) are possible. Furthermore, in order to make it a low-water-flow daily dose, the equipment which carries out circulation purification of the environmental gas is installed separately, there is the approach of carrying out circulation operation, and it is possible according to this approach to maintain -100 degrees C (about 0.06 ppm) of below dew-point abbreviation.

[0053] Thermosetting resin, a photo-setting resin, etc. are used as a sealing compound 6. Also in which resin, compared with acrylic and a silicon system, epoxy system resin is excellent in respect of moisture permeability, and, generally is used well. The thickness of a sealing compound 6 needs to be thicker than the thickness of the sum total of the component (stratified deposit) formed in the substrate 1. For this reason, a silica bead, glass fiber, etc. may be used together with a sealing compound as a spacer in order to secure the thickness of the seal section.

[0054] Moreover, as a sealing agent on the back, although a metal, resin, etc. may be used other than tooth-back glass 7, also in which sealing agent, moisture permeability-proof, oxygen-proof permeability, etc. are required like a sealing compound. A sealing agent also takes into consideration properties, such as the visible permeability of a tooth-back sealing agent, reinforcement, and variability, and selection use is carried out again according to the application of a component.

[0055] Next, with reference to drawing 1, the manufacture approach of the organic electroluminescence devices by the manufacturing installation and this equipment of organic electroluminescence devices of this invention for manufacturing such organic electroluminescence devices is explained.

[0056] Drawing 1 is the mimetic diagram showing the gestalt of operation of the manufacturing installation of the organic electroluminescence devices of this invention.

[0057] They are the gate valve with which the vacuum chamber for conveyance, and 22b-26b connect a substrate and the robot arm for mask conveyance, and, as for a dry box, and 22-26, 22c-27c connect a dry box, the vacuum chamber for conveyance, or the vacuum chambers for conveyance for 21 and 27, as for a working-level month vacuum chamber, and 22a-26a, and the gate valve which connects a working-level month vacuum chamber and the vacuum chamber for conveyance 22d-26d among drawing 1.

[0058] After installing that by which the anode plate 2 by which patterning was carried out was formed on the substrate 1 in a dry box 21 in manufacture of organic electroluminescence devices and carrying out UV ozone washing of the substrate front face, inert gas fully permutes the inside of a dry box 21. Next, gate valve 22c is opened and a substrate is introduced in vacuum chamber 22a for conveyance using robot arm 22b. After closing gate valve 22c, vacuum suction of the vacuum chamber 22a for conveyance is carried out to 10 to 5 or less Toors, gate valve 22d is opened after that, a substrate is transported using robot arm 22b, and a substrate is installed in the working-level month vacuum chamber 22. By the working-level month vacuum chamber 22, plasma treatment of the substrate front face is carried out by the mixed gas of an argon and oxygen. Next, a substrate is transported through gate valve 22d and vacuum chamber 22 for conveyance a, gate valve 23c, vacuum chamber 23 for conveyance a, and gate valve 23d, using the robot arms 22b and 23b one by one, with a vacuum held, and a substrate is installed in the working-level month vacuum chamber 23. Within the working-level month vacuum chamber 23, sequential vacuum evaporatio formation is carried out, and hole injection layer 3c and electron hole transportation layer 3a transport a substrate like the above after that, using the robot arms 23b and 24b one by one, and install a substrate in the working-level month vacuum chamber

24. Within the working-level month vacuum chamber 24, vacuum evaporation formation is carried out, and electronic transportation layer 3b transports a substrate like the above after that, using the robot arms 24b and 25b one by one, and installs a substrate in the working-level month vacuum chamber 25. Within the working-level month vacuum chamber 25, vacuum evaporation formation of the cathode 4 is carried out, a substrate is transported like the above after that, using the robot arms 25b and 26b one by one, and a substrate is installed in the working-level month vacuum chamber 26. Within the working-level month vacuum chamber 26, vacuum evaporation formation is carried out, and a protective layer 5 installs a substrate in vacuum chamber 26a for conveyance by robot arm 26b after that, closes gate valve 26d, and is filled up with inert gas to atmospheric pressure in vacuum chamber 26a for conveyance.

[0059] Next, gate valve 27c is opened, in the dry box 27 beforehand filled with inert gas, robot arm 26b is used and a substrate is installed. In a dry box 27, after applying the photo-setting resin of a sealing compound 6 on a substrate 1, tooth-back glass 7 is made to contact and the seal of the organic electroluminescence-devices body 10B is carried out.

[0060] As mentioned above, according to this equipment, a substrate is not exposed to atmospheric air after filling up with the inert gas after UV ozone washing in a dry box 21 until a seal is completed in a dry box 27.

[0061] Thus, it is taken out from a dry box 27, the electric wiring for a component drive and mounting of a semiconductor circuit are made, and the organic electroluminescence devices which the seal completed are completed as a panel.

[0062] In addition, although the unit for migration and processing which consists of a working-level month vacuum chamber, a vacuum chamber for conveyance, etc. is connected with the five-piece serial in the shape of a straight line in drawing 1 This number of connection units does not necessarily need to be five pieces, and it can also perform easily being able to connect the number of two or more arbitration by the class of the number of stratified deposits, or stratified deposit, the other arts of a substrate, etc., and fluctuating the number of units according to the change in a stratified deposit, modification of an art, etc. Moreover, two dry boxes are not necessarily required, either and it is also possible to collect two functions to one or to omit one function. A robot arm needs a certain thing for one or more vacuum chambers for conveyance, and when two or more [of these] are prepared, migration of a substrate and a mask becomes efficient and it can shorten working hours more.

[0063] A robot arm here points out the thing of the device which holds a substrate and a mask and can move between vacuum chambers, the maintenance means or migration means of the configuration of equipment and a substrate is not specified at all, and combination with a conveyor type migration means etc. is also possible.

[0064] Moreover, the processing in the working-level month vacuum chambers 22-26 is not limited to the approach explained above at all, but can adopt various arts, such as a spatter and an electron beam method, as the membrane formation approach. Moreover, ultraviolet rays and an excimer laser can be irradiated in addition to the plasma treatment method according to the mixed gas of argon gas and oxygen gas also as an art of the substrate in the working-level month vacuum chamber 22, or various substrate arts, such as the approach of exposing to specific gas and reforming a substrate front face, can be adopted. For example, in the working-level month vacuum chamber 22, the plasma or laser radiation performs reforming of a substrate, in the working-level month vacuum chambers 23-25, with the vacuum deposition by resistance heating, each organic layer and cathode can be formed and a protective layer can also be formed by the spatter in the working-level month vacuum chamber 26. Moreover, in one working-level month vacuum chamber, it can also have two or more approaches, each approach is used, and the membrane formation approach and a substrate art can also be formed membranes or processed to sequential or coincidence. Moreover, although the art after carrying out pattern formation of the anode plate as a lower electrode here was indicated, it is also possible to also form a lower electrode in the equipment of illustration. Moreover, the order of formation of an anode plate and cathode was not fixed, either, and it can choose freely to compensate for the configuration of an organic layer.

[0065] This invention is applicable also to a single component, the component which consists of

structure arranged in the shape of an array, and which organic electroluminescence devices of the structure where an anode plate and cathode have been arranged in the shape of an X-Y matrix.

[0066]

[Example] Next, although an example and the example of a comparison are given and this invention is explained still more concretely, this invention is not limited to the publication of the following examples, unless the summary is exceeded.

[0067] Using 7059 by Corning, Inc. glass with a thickness of 1.1mm as example 1 glass substrate, 120nm deposition (Geomatec electron beam membrane formation article; 20ohms of sheet resistance) of the indium stannic-acid ghost (ITO) transparence electric conduction film was carried out on it, and the glass substrate with the ITO film was obtained.

[0068] Next, the organic electroluminescence devices which have the structure shown in drawing 5 using the equipment shown in drawing 1 were produced by the following approaches.

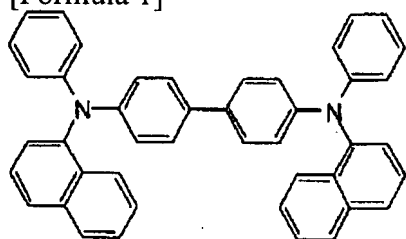
[0069] Patterning of the ITO transparence electric conduction film deposited on the glass substrate 1 was carried out to the stripe of 2mm width of face using the usual photolithography technique and hydrochloric-acid etching, and the anode plate 2 was formed. It was made to dry by nitrogen blow after washing in order of ultrasonic cleaning by the acetone, rinsing by pure water, and ultrasonic cleaning by isopropyl alcohol, and the ITO substrate which carried out pattern formation was installed in the dry box 21. In the dry box 21, after performing UV / ozone washing for 10 minutes, inert gas replacement of the inside of a dry box was carried out with nitrogen gas, gate valve 22c was opened, and the substrate was introduced in vacuum chamber 22a for conveyance by robot arm 22b. After closing gate valve 22c, vacuum suction of the inside of vacuum chamber 22a for conveyance was carried out also using cryopump to 1.1×10^{-6} Torr (about 1.5×10^{-3} to 4 Pa). Then, gate valve 22d was opened and the substrate 1 was installed in the working-level month vacuum chamber 22 using robot arm 22b. After closing gate valve 22d, in the working-level month vacuum chamber 22 which was the vacua of 1.0×10^{-6} Torr, mixed gas of 50% of oxygen was introduced argon 50%, until it was set to 1.0×10^{-3} Torr, and the front face of the glass substrate 1 with ITO was processed for 5 minutes in the plasma. Installation of the blasting fumes was suspended, and vacuum suction of the inside of the working-level month vacuum chamber 22 was carried out until it would be in the vacua of 1.0×10^{-6} Torr again.

[0070] Next, gate valve 22d was opened, the substrate 1 was transported into vacuum chamber 22a for conveyance from the working-level month vacuum chamber 22 using robot arm 22b, after closing gate valve 22d, gate valve 23c was opened, the substrate 1 was transported into vacuum chamber 23a for conveyance using robot arm 22b and robot arm 23b, and gate valve 23c was closed. While the substrate 1 was transported to 23a from vacuum chamber 22 for conveyance a, the inside of vacuum chamber 22 for conveyance a and 23a was maintained at the vacua of 1.0×10^{-6} Torr.

[0071] Next, gate valve 23d was opened and the substrate 1 was installed in the working-level month vacuum chamber 23 using robot arm 23b. Under the present circumstances, a substrate 1 is installed on the metal mask arranged beforehand, and the part for the anode plate ejection on a substrate 1 was covered with the metal mask. Next, gate valve 23d was closed and it vapor-deposited by heating the copper phthalocyanine (H1) (crystal form being beta mold) shown in the following put into the molybdenum boat arranged in the working-level month vacuum chamber 23. It vapor-deposited in degree of vacuum 1.1×10^{-6} Torr (about 1.5×10^{-3} to 4 Pa), and vacuum evaporation time amount 1 minute, and hole injection layer of 20nm of thickness 3c was obtained.

[0072]

[Formula 1]

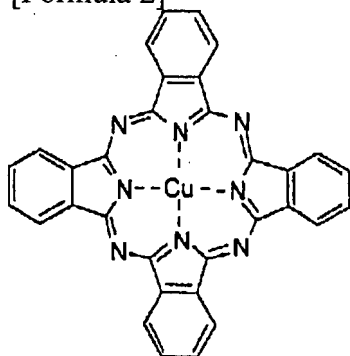


(H 1)

[0073] Next, 4 and 4' bis[-] [N-(1-naphthyl)-N-phenylamino] biphenyl (H2) shown in the following put into the ceramic crucible arranged in the working-level month vacuum chamber 23 was heated at the tantalum wire heater around a crucible, and the laminating was carried out on hole injection layer 3c. The temperature of the end crater which gets at this time was controlled in 230-240 degrees C. Electron hole transportation layer of 60nm of thickness 3a was obtained in degree of vacuum 8×10^{-7} Torr at the time of vacuum evaporation (about 1.1×10 to 4 Pa), and vacuum evaporation time amount 50 seconds per minute.

[0074]

[Formula 2]

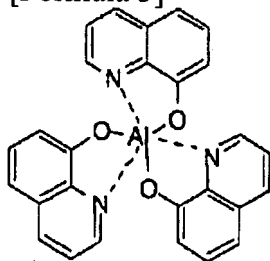


(H 2)

[0075] Next, gate valve 23d was opened, the substrate 1 was transported into vacuum chamber 23a for conveyance from the working-level month vacuum chamber 23 using robot arm 23b, after closing gate valve 23d, gate valve 24c was opened, the substrate 1 was transported into vacuum chamber 24a for conveyance using robot arm 23b and robot arm 24b, and gate valve 24c was closed. Then, gate valve 24d was opened and the substrate 1 was installed in the working-level month vacuum chamber 24 using robot arm 24b. Under the present circumstances, a substrate 1 is installed on the metal mask arranged beforehand, and the part for the anode plate ejection on a substrate 1 was covered with the metal mask. Next, gate valve 24d was closed and 8-hydroxyquinoline complex $\text{Al}(\text{C}_9\text{H}_6\text{NO})_3$ (E1) of the aluminum shown with the following structure expressions as an ingredient of electronic transportation layer 3b which has a luminescence function within the working-level month vacuum chamber 24 was vapor-deposited similarly on the above-mentioned electron hole transportation layer 3a. The temperature of the end crater which gets at this time was controlled in 310-320 degrees C. The degree of vacuum at the time of vacuum evaporation was 9×10^{-7} Torr (about 1.2×10 to 4 Pa), vacuum evaporation time amount was 2 minutes and 40 seconds, and the thickness of vapor-deposited electronic transportation layer 3b was 75nm.

[0076]

[Formula 3]



(E 1)

[0077] In addition, the substrate temperature when carrying out vacuum deposition of the above-mentioned hole injection layer 3c, electron hole transportation layer 3a, and the electronic transportation layer 3b was held to the room temperature.

[0078] Next, gate valve 24d was opened, the substrate 1 was transported into vacuum chamber 24a for conveyance from the working-level month vacuum chamber 24 using robot arm 24b, after closing gate

valve 24d, gate valve 25c was opened, the substrate 1 was transported into vacuum chamber 25a for conveyance using robot arm 24b and robot arm 25b, and gate valve 25c was closed. Gate valve 25d was opened after that, and the substrate 1 was installed in the working-level month vacuum chamber 25 using robot arm 25b. Under the present circumstances, a substrate 1 is installed on the metal mask for cathode vacuum evaporation arranged beforehand, and the stripe-like shadow mask of 2mm width of face was stuck to the substrate 1 so that it might intersect perpendicularly with the ITO stripe of the patternized anode plate 2.

[0079] Next, gate valve 25d was closed, and within the working-level month vacuum chamber 25, as cathode 4, the alloy electrode of magnesium and silver was vapor-deposited so that 2 μm might become 100nm of thickness with coincidence vacuum deposition. Vacuum evaporation was performed using the molybdenum boat in degree of vacuum 1×10^{-5} Torr (about 1.3×10 to 3 Pa), and vacuum evaporation time amount 3 minutes and 10 seconds. Moreover, the atomic ratio of magnesium and silver was set to 10:1.2. Furthermore it continued, the laminating of the aluminum was carried out on magnesium and the silver alloy film by 100nm thickness using the molybdenum boat into the working-level month vacuum chamber 25, and cathode 4 was completed. The degree of vacuum at the time of the vacuum plating of aluminium was 2.3×10^{-5} Torr (about 3.1×10 to 3 Pa), and vacuum evaporation time amount was 40 seconds per minute. The substrate temperature at the time of vacuum evaporation of the above magnesium and silver alloy, and the two-layer mold cathode of aluminum was held to the room temperature.

[0080] Next, gate valve 25d was opened, the substrate 1 was transported into vacuum chamber 25a for conveyance from the working-level month vacuum chamber 25 using robot arm 25b, after closing gate valve 25d, gate valve 26c was opened, the substrate 1 was transported into vacuum chamber 26a for conveyance using robot arm 25b and robot arm 26b, and gate valve 26c was closed. Gate valve 26d was opened after that, and the substrate 1 was installed in the working-level month vacuum chamber 26 using robot arm 26b. Under the present circumstances, the substrate 1 was stuck and installed on the metal mask for protective layer vacuum evaporation arranged beforehand. Next, gate valve 26d was closed, and within the working-level month vacuum chamber 26, as a protective layer 5, silicon oxide ($\text{SiO}_x; x=1.0-2.0$) was vapor-deposited so that it might become 1000nm of thickness. Vacuum evaporation was performed using the molybdenum boat in degree of vacuum 1×10^{-5} Torr (about 1.3×10 to 3 Pa), and vacuum evaporation time amount 3 minutes and 40 seconds. The substrate temperature at the time of this vacuum evaporation was held to the room temperature. Thereby, the component section except a part for the takeoff connection of an anode plate and cathode was covered by the protective layer 5.

[0081] Next, after having opened gate valve 26d, transporting the substrate 1 into vacuum chamber 26a for conveyance from the working-level month vacuum chamber 26 using robot arm 26b and closing gate valve 26d, nitrogen was introduced in vacuum chamber 26a for conveyance, and it considered as atmospheric pressure.

[0082] Then, gate valve 27c was opened, in the dry box 27 currently beforehand filled with nitrogen gas, robot arm 26b was used, and the substrate 1 was installed, next dispensing of the photo-setting resin (30Y-184) by Three Bond Co., Ltd. was carried out to the seal section on the substrate 1 of organic electroluminescence-devices body 10B by width of face of 0.5mm as a sealing compound 6. Then, they are 50 g/cm² so that a sealing compound 6 may install the tooth-back glass 7 beforehand cut into the size of the seal section in the part carried out with ** and a glass substrate 1 and tooth-back glass 6 may stick it. The pressure was put for 30 seconds.

[0083] Next, it is ultraviolet radiation (wavelength of 365nm) to the part to which the sealing compound 6 was carried out with ** 4.2 J/cm² It irradiated, the photo-setting resin was stiffened and the seal was made to complete. On the occasion of hardening, in order to prevent degradation of the component by ultraviolet rays and heat, parts other than the seal section shaded.

[0084] Then, the component was taken out from the ejection room established in the dry box 27, and the organic electroluminescence devices of 2mmx2mm size were obtained.

[0085] Impress the direct current voltage of minus, it was made to emit light to the anode plate 2 of the

obtained organic electroluminescence devices in plus and cathode 4, and the luminescence property was measured. Current density 15 mA/cm² The time amount change to the initial value of the luminescence brightness when continuing passing a current for a component is shown in drawing 6.

[0086] The luminescence brightness in early stages of [energization] the organic electroluminescence devices by this example was 9250 cd/m². The luminescence brightness after 10000-hour progress is 8650 cd/m². It became, the relative luminance to initial value was 0.935, and the fall of brightness was small.

[0087] The organic electroluminescence devices of the same lamination as the organic electroluminescence devices manufactured in the example 1 for the example of comparison 1 comparison were produced by the option.

[0088] That is, except for having once taken out the substrate 1 from the working-level month vacuum chamber into atmospheric air 2 times after formation of a protective layer 5 the formation back of electronic transportation layer 3b, and having moved to the following process using the isolated system, others produced organic electroluminescence devices on the same conditions as an example 1, without two or more working-level month vacuum chambers connecting.

[0089] The configuration, the quality of the material, the thickness, and the appearance of a layer of the obtained organic electroluminescence devices were the same as that of the thing of an example 1.

[0090] About these organic electroluminescence devices, luminescence brightness was measured by the same approach as an example 1, and the time amount change to the initial value of luminescence brightness was shown in drawing 6.

[0091] the organic electroluminescence devices of this example of a comparison -- the luminescence brightness in early stages of energization -- 9100 cds/m² it is -- there were not a thing of an example 1 and a big difference. However, the luminescence brightness after 1000-hour progress is 5105 cd/m². The relative luminance to initial value is set to 0.561, and 10000 hours after, it will not shine, so that measurement of luminescence brightness is impossible.

[0092] The organic electroluminescence devices of the same lamination as the organic electroluminescence devices manufactured in the example 1 for the example of comparison 2 comparison were further produced by the option.

[0093] That is, except for having once taken out the substrate 1 from the working-level month vacuum chamber into atmospheric air after formation of a protective layer 5, and having moved to the following seal process using the equipment with which two or more working-level month vacuum chambers were connected with the perimeter of one vacuum chamber for conveyance, others produced organic electroluminescence devices on the same conditions as an example 1.

[0094] The configuration, the quality of the material, the thickness, and the appearance of a layer of the obtained organic electroluminescence devices were the same as that of the thing of an example 1.

[0095] About organic electroluminescence devices, luminescence brightness was measured by the same approach as an example 1, and the time amount change to the initial value of luminescence brightness was shown in drawing 6.

[0096] the organic electroluminescence devices of this example of a comparison -- the luminescence brightness in early stages of energization -- 9150 cds/m² it is -- there were not a thing of an example 1 and a big difference. However, the luminescence brightness after 10000-hour progress is 6680 cd/m². Although the relative luminance to initial value was set to 0.73 and its fall of brightness was smaller than the example 1 of a comparison, compared with the example 1, its fall of brightness was large.

[0097]

[Effect of the Invention] According to this invention, each class-like deposit prevents contacting the moisture and oxygen in atmospheric air by carrying out without being consistent also including each process and its shift process from after the pattern formation of an anode plate to sealing, and exposing a substrate to atmospheric air in manufacture of organic electroluminescence devices, productivity is good and organic electroluminescence devices excellent in the luminescence property and the luminescence life property can be offered by low cost as explained in full detail above.

[0098] The application to the light source (for example, the light source of a copying machine, the back

light light source of a performance display or instruments) and the plotting board with which the organic electroluminescence devices manufactured by this invention employed the description as a flat-panel display (for example, the object for OA computers and a flat TV) or a field illuminant efficiently, and a beacon light is expected, and the technical value is very large.

[Translation done.]

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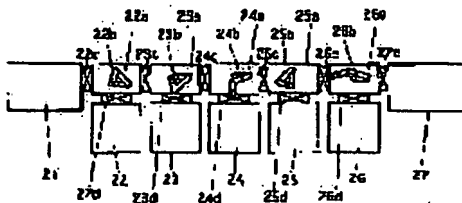
SATO YOSHIHARU

(54) MANUFACTURING DEVICE AND MANUFACTURE OF ORGANIC ELECTROLUMINESCENT ELEMENT

(57)Abstract:

PROBLEM TO BE SOLVED: To prevent respective layer-shaped deposits from contacting with moisture and oxygen in the atmosphere, and inexpensively manufacture an organic electroluminescence element excellent in a light emitting service life characteristic by consistently processing a base board without being exposed to the atmosphere in respective processes and a transfer process up to sealing after a pattern of an anode is formed.

SOLUTION: A base board on which an anode is formed as a pattern is arranged in a dry box 21, and after a base board surface is cleaned, it is sufficiently substituted with inert gas.



Afterwards, the base board is carried and processed in order in working vacuum chambers 22 to 26 by respective prescribed operations of gate valves 22c to 27c and

22d to 26d, carrying vacuum chambers 22a to 26a and base board and mask carrying robot arms 22b to 26b. After respective layers are formed on the base board by these processings, the base board is not exposed to the atmosphere until a seal is completed by the dry box 27 filled with inert gas. Therefore, an organic electroluminescence element excellent in a light emitting characteristic and a light emitting service life characteristic can be inexpensively manufactured with excellent productivity.

*** NOTICES ***

JPO and NCIP are not responsible for any damages caused by the use of this translation.

1. This document has been translated by computer. So the translation may not reflect the original precisely.

2. **** shows the word which can not be translated.

3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]

[Claim 1] In the equipment for manufacturing the organic electroluminescence devices which come to prepare two or more stratified deposits on a substrate The vacuum chamber for conveyance with carrying-in opening and taking-out opening of a substrate, and the working-level month vacuum chamber which stands in a row through the bulb which can pass [that vacuum transfer cutoff is possible and] said substrate in this vacuum chamber for conveyance, A migration means to be installed in this vacuum chamber for conveyance, and to transport a substrate into this working-level month vacuum chamber from said carrying-in opening, and to transport to said taking-out opening from this working-level month vacuum chamber, It is the organic electroluminescence-devices manufacturing installation equipped with the 1st thru/or the n-th unit for migration and processing which has this processing means for stratified deposit formation established in this working-level month vacuum chamber. So that the substrate introduced in this manufacturing installation may be transported from carrying-in opening of the 1st unit to the n-th unit through this unit The manufacturing installation of the organic electroluminescence devices characterized by connecting taking-out opening of the unit of the substrate migration direction upstream to carrying-in opening of the unit of the substrate migration direction downstream.

[Claim 2] The manufacturing installation of the organic electroluminescence devices which are equipment for manufacturing the organic electroluminescence devices which come to prepare the seal member surrounding this stratified deposit while preparing two or more stratified deposits on a substrate, and are characterized by to have the facility for preparing said seal member, without exposing to atmospheric air after forming all stratified deposits in the manufacturing installation of the organic electroluminescence devices which prepared the following stratified deposit, without exposing to atmospheric air after preparing one stratified deposit.

[Claim 3] The manufacturing installation of the organic electroluminescence devices characterized by the equipment for preparing said stratified deposit on a substrate being a manufacturing installation according to claim 1 in claim 2.

[Claim 4] The manufacture approach of the organic electroluminescence devices characterized by to prepare said seal member, without exposing to atmospheric air after forming in the approach of manufacturing the organic electroluminescence devices which come to have the stratified deposit by which deposition formation was carried out, and which has an anode plate, an organic luminous layer, and cathode at least, and a seal member surrounding this stratified deposit to the shape of a layer on a substrate and this substrate, without exposing an organic luminous layer and cathode to atmospheric air within a vacuum tub at least among these stratified deposits.

[Translation done.]

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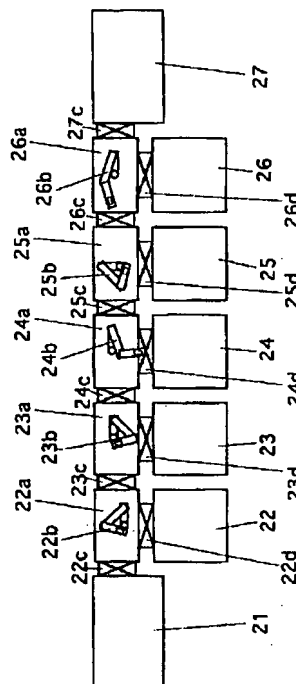
(54)【発明の名称】 有機電界発光素子の製造装置及び製造方法

(57)【要約】

【課題】 発光寿命特性に優れた有機電界発光素子を量産性良く、低コストで効率的に製造する。

【解決手段】 作業用真空室22とロボットアーム22bを備える搬送用真空室22aとが、ゲートバルブ22dで連結された移送及び蒸着用ユニットをゲートバルブ23cで複数個連結した有機電界発光素子の製造装置。この製造装置により、基板を大気に晒すことなく、基板上に有機発光層を含む層状堆積物を形成しかつシーリングを行う。

【効果】 有機電界発光素子を陽極のパターン形成後からシーリングまでの各工程及びその移行工程も含めて一貫して基板を大気に晒すことなく実施することで、各層状堆積物が大気中の水分や酸素に接触するのを防止して、発光寿命特性に優れた有機電界発光素子を生産性良く、かつ低コストで製造できる。ユニットの増設も容易に行える。



【特許請求の範囲】

【請求項1】 基板上に複数の層状堆積物を設けてなる有機電界発光素子を製造するための装置において、基板の搬入口及び搬出口を有した搬送用真空室と、該搬送用真空室内に真空伝達遮断可能かつ前記基板が通過可能なバルブを介して連なっている作業用真空室と、該搬送用真空室内に設置されており、基板を、前記搬入口から該作業用真空室内に移送し、かつ該作業用真空室から前記搬出口へ移送する移送手段と、該作業用真空室内に設けられた、該層状堆積物形成用処理手段と、を有する移送及び処理用の第1ないし第nのユニットを備えた有機電界発光素子製造装置であって、第1のユニットの搬入口から該製造装置内に導入された基板が該ユニットを通過して第nのユニットまで移送されるように、基板移送方向上流側のユニットの搬出口が基板移送方向下流側のユニットの搬入口に対し接続されていることを特徴とする有機電界発光素子の製造装置。

【請求項2】 基板上に複数の層状堆積物を設けると共にこの層状堆積物を囲むシール部材を設けてなる有機電界発光素子を製造するための装置であって、1つの層状堆積物を設けた後、大気に晒すことなく次の層状堆積物を設けるようにした有機電界発光素子の製造装置において、すべての層状堆積物を形成した後、大気に晒すことなく前記シール部材を設けるための設備を備えたことを特徴とする有機電界発光素子の製造装置。

【請求項3】 請求項2において、前記層状堆積物を基板上に設けるための装置が請求項1に記載の製造装置であることを特徴とする有機電界発光素子の製造装置。

【請求項4】 基板と、該基板上に層状に堆積形成された少なくとも陽極、有機発光層及び陰極を有する層状堆積物と、該層状堆積物を囲むシール部材とを備えてなる有機電界発光素子を製造する方法において、該層状堆積物のうち少なくとも有機発光層及び陰極を真空槽内で大気に晒すことなく形成した後、大気に晒すことなく前記シール部材を設けることを特徴とする有機電界発光素子の製造方法。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は有機電界発光素子の製造装置及びその製造方法に関するものであり、詳しくは、有機化合物から成る発光層に電界をかけて光を放出する薄膜型発光素子を製造する装置及び方法であって、発光寿命特性に優れた有機電界発光素子を量産性良く、低コストで効率的に製造する装置及び方法に関するものである。

【0002】

【従来の技術】有機電界発光素子は、自発光、薄型、高視野角などの特徴を有し、新しい平面型発光源ないし表示素子として注目されている。

【0003】従来、有機電界発光素子を製造するためには、ガラス基板上にITO（インジウム・スズ酸化物）などの透明導電膜をスパッタ等の方法で形成した後、パターン加工して下部電極を形成し、この基板を真空蒸着槽内に設置して加熱蒸着などの方法で有機発光層等を形成した後、該真空槽からいったん取り出して別の真空槽で金属層や保護層等を形成し、その後この真空槽から取り出して素子部のシーリングを行っていた。

【0004】上記従来の方法では、有機正孔輸送層、有機発光層、陰極、保護層等の多層構造を形成するために、真空槽をその都度真空から常圧に戻して、原料を入れ替えたりマスクを交換したりする必要があった。また、真空槽を複数用意した場合でも、真空槽間の基板の入れ替えが必要であった。このため、先の蒸着と次の蒸着との間で基板が大気に晒され、蒸着層が大気中の酸素や水分に暴露されるため、素子の特性が低下するという問題があった。また、真空槽の真空引きと常圧解放を繰り返すため、製造時間が長く、生産効率が悪いという問題点もあった。

【0005】これらの問題点を解決する方法として、透明導電膜のパターン加工後から保護層の形成までの真空蒸着工程を一貫して行うことを目的に、1つの真空搬送槽を中心とし、その周りに真空蒸着槽を複数個配置して、その間を真空状態を保ったまま基板の受け渡しをして順次蒸着する方法が試みられている（特開平8-111285号公報）。

【0006】

【発明が解決しようとする課題】しかしながら、この方法では、搬送槽の周りに配置できる蒸着槽の数に限りがある；基板サイズが大きくなると特に搬送槽が巨大化する構造上の特徴から、量産機には向かない；基板は、保護層形成後、シール工程の前に真空槽から外に排出され、大気に晒されてしまうため、素子の長寿命化が難しい；等の問題点があった。

【0007】本発明者は上記従来の問題点を解決し、特性の優れた有機電界発光素子を低コストで量産性良く、製造することができる装置及び方法を提供することを目的とする。

【0008】

【課題を解決するための手段】請求項1の有機電界発光素子の製造装置は、基板上に複数の層状堆積物を設けてなる有機電界発光素子を製造するための装置において、基板の搬入口及び搬出口を有した搬送用真空室と、該搬送用真空室内に真空伝達遮断可能かつ前記基板が通過可能なバルブを介して連なっている作業用真空室と、該搬送用真空室内に設置されており、基板を、前記搬入口から該作業用真空室内に移送し、かつ該作業用真空室から前記搬出口へ移送する移送手段と、該作業用真空室内に設けられた、該層状堆積物形成用処理手段と、を有する移送及び処理用の第1ないし第nのユニットを備えた有

機電界発光素子製造装置であって、第1のユニットの搬入口から該製造装置内に導入された基板が該ユニットを
通って第nのユニットまで移送されるように、基板移送
方向上流側のユニットの搬出口が基板移送方向下流側の
ユニットの搬入口に対し接続されていることを特徴とす
る。

【0009】この有機電界発光素子の製造装置であ
れば、第1のユニットの搬送用真空室、第1のユニットの
作業用真空室、第1のユニットの搬送用真空室、第2の
ユニットの搬送用真空室、第2のユニットの作業用真空
室及び第2のユニットの搬送用真空室、……第(n-
1)のユニットの搬送用真空室、第nのユニットの搬送
用真空室、第nのユニットの作業用真空室、第nのユニ
ットの搬送用真空室の順で基板を順次移送することによ
り、基板を大気に晒すことなく、真空状態を維持して層
状堆積物を効率的に形成することができる(なお、任意
のユニットの作業用真空室を経由せずに層状堆積物を形
成していくことも可能であるし、あるいは、例えば第3
のユニットから第2のユニットに戻って層状堆積物を形
成することも可能である)。

【0010】しかも、この移送及び処理用のユニット
は、搬送用真空室により接続されているため、任意の数
を連結することができ、層状堆積物を形成するための作
業用真空室数が制限されることはない。このユニットの
増設も容易で蒸着する層状堆積物の増加にも容易に対応
でき、製造コストを抑えることができる。また、基板サ
イズが大きい場合でも、搬送用真空室が巨大化すること
もない。

【0011】請求項2の有機電界発光素子の製造装置
は、基板上に複数の層状堆積物を設けると共にこの層状
堆積物を囲むシール部材を設けてなる有機電界発光素子
を製造するための装置であって、1つの層状堆積物を設
けた後、大気に晒すことなく次の層状堆積物を設けるよ
うにした有機電界発光素子の製造装置において、すべて
の層状堆積物を形成した後、大気に晒すことなく前記シ
ール部材を設けるための設備を備えたことを特徴とす
る。

【0012】この有機電界発光素子の製造装置であ
れば、基板に層状堆積物を形成した後、この層状堆積物
を囲むシール部材を、基板を大気に晒すことなく設ける
ことができ、発光寿命特性に優れた有機電界発光素子を製
造することができる。

【0013】この装置において、基板上に層状堆積物
を設けるための装置は前記請求項1に記載の製造装置であ
ることが好ましい。

【0014】請求項4の有機電界発光素子の製造方法
は、基板と、該基板上に層状に堆積形成された少なくと
も陽極、有機発光層及び陰極を有する層状堆積物と、該
層状堆積物を囲むシール部材とを備えてなる有機電界発
光素子を製造する方法において、該層状堆積物のうち少

なくとも有機発光層及び陰極を真空槽内で大気に晒すこ
となく形成した後、大気に晒すことなく前記シール部材
を設けることを特徴とする。

【0015】この方法では、層状堆積物の形成からシ
ール部材の形成まで、一貫して基板を大気に晒すことな
く行うため、著しく発光特性及び発光寿命特性に優れた有
機電界発光素子を製造することができる。

【0016】

【発明の実施の形態】以下に図面を参照して本発明の実
施の形態を詳細に説明する。

【0017】まず、図2〜5を参照して本発明で製造さ
れる有機電界発光素子の構成を説明する。

【0018】図2〜4は本発明で製造される有機電界発
光素子の有機電界発光素子本体の構造例を示す模式的な
断面図であり、図5はこのような有機電界発光素子本体
をシール部材でシーリングしてなる有機電界発光素子を
示す模式的な断面図である。

【0019】図2〜5において、1は基板、2は陽極、
3は有機発光層、3aは正孔輸送層、3bは電子輸送
層、3cは正孔注入層、4は陰極、5は保護層、6はシ
ール剤、7は背面ガラス、10、10A、10Bは有機
電界発光素子本体である。

【0020】基板1は有機電界発光素子の支持体となる
ものであり、光学特性、耐熱性、表面精度、機械的強
度、軽量性、ガスバリア性などの特性に優れていること
が要求される。基板1としては、一般に、石英やガラス
の板、金属板や金属箔、プラスチックフィルムやシート
などが用いられるが、ガラス板や、ポリエステル、ポリ
メタアクリルレート、ポリカーボネート、ポリサルホン
などの透明な合成樹脂基板が好適である。

【0021】基板1上に形成された陽極2は、有機発光
層3への正孔注入の役割を果たすものである。この陽極
2は、通常、アルミニウム、金、銀、白金、ニッケル、
パラジウム、白金等の金属、インジウム及び/又はスズ
の酸化物などの金属酸化物、ヨウ化銅などのハロゲン化
金属、カーボンブラック、或いは、ポリ(3-メチルチ
オフエン)、ポリピロール、ポリアニリン等の導電性高
分子など、好ましくは、インジウム・スズ酸化物(ITO)
により形成される。陽極2の形成は通常、スパッタ
リング法、真空蒸着法などにより行われることが多い。
銀などの金属微粒子、ヨウ化銅などの微粒子、カーボン
ブラック、導電性の金属酸化物微粒子、導電性高分子微
粉末などを用いる場合には、これを適当なバインダー樹
脂溶液に分散し、基板1上に塗布することにより陽極2
を形成することもできる。また、導電性高分子を用いる
場合には、電解重合により直接基板1上に薄膜を形成す
るか、基板1上に導電性高分子を塗布することにより、
陽極2を形成することもできる。陽極2は異なる物質を
積層して形成することも可能である。

【0022】陽極2の厚みは、透明性の要求の有無によ

り異なる。透明性が必要とされる場合は、可視光の透過率を、通常、60%以上、好ましくは80%以上とすることが望ましく、この場合、厚みは、通常、5~1000nm、好ましくは10~500nm程度である。陽極2が不透明でよい場合には、基板1と同一材料であってもよい。また、陽極2の上に異なる導電材料を積層することも可能である。

【0023】陽極2の上に形成される有機発光層3は、電界が与えられた電極間において、陽極2から注入された正孔と陰極4から注入された電子を効率よく輸送して再結合させ、かつ、再結合により効率よく発光する材料から形成される。通常、この有機発光層3は発光効率の向上のために、図3に示す様に、正孔輸送層3aと電子輸送層3bに分割した機能分離型にすることが行われる。

【0024】図3に示す機能分離型有機電界発光素子10Aにおいて、正孔輸送層3aの材料としては、陽極2からの正孔注入効率が高く、かつ、注入された正孔を効率よく輸送することができる材料であることが必要である。そのためには、イオン化ポテンシャルが小さく、しかも正孔移動度が大きく、更に安定性に優れ、製造時や使用時にトラップとなる不純物が発生しにくいことが要求される。

【0025】このような正孔輸送材料としては、例えば、1, 1-ビス(4-ジ-*p*-トリルアミノフェニル)シクロヘキサン等の3級芳香族アミンユニットを連結した芳香族ジアミン化合物、4, 4'-ビス[N-(1-ナフチル)-N-フェニルアミノ]ビフェニルで代表される2個以上の3級アミンを含み2個以上の縮合芳香族環が窒素原子に置換した芳香族アミン、トリフェニルベンゼンの誘導体でスターバースト構造を有する芳香族トリアミン、N, N'-ジフェニル-N, N'-ビス(3-メチルフェニル)ビフェニル-4, 4'-ジアミン等の芳香族ジアミン、 $\alpha, \alpha, \alpha', \alpha'$ -テトラメチル- α, α' -ビス(4-ジ-*p*-トリルアミノフェニル)-*p*-キシレン、分子全体として立体的に非対称なトリフェニルアミン誘導体、ビレニル基に芳香族ジアミノ基が複数個置換した化合物、エチレン基で3級芳香族アミンユニットを連結した芳香族ジアミン、スチリル構造を有する芳香族ジアミン、チオフェン基で芳香族3級アミンユニットを連結したもの、スターバースト型芳香族トリアミン、ベンジルフェニル化合物、フルオレン基で3級アミンを連結したもの、トリアミン化合物、ビスピリジルアミノビフェニル、N, N, N'-トリフェニルアミン誘導体、フェノキサジン構造を有する芳香族ジアミン、ジアミノフェニルフェナントリジン誘導体、ヒドラゾン化合物、シラザン化合物、シラナミン誘導体、ホスファミン誘導体、キナクリドン化合物等が挙げられる。これらの化合物は、単独で用いてもよく、また、必要に応じて2種以上を混合して用いてもよい。

【0026】なお、上記の化合物以外に、正孔輸送層3aの材料として、ポリビニルカルバゾールやポリシラン、ポリフォスフェゼン、ポリアミド、ポリビニルトリフェニルアミン、トリフェニルアミン骨格を有する高分子、トリフェニルアミン単位をメチレン基等で連結した高分子、芳香族アミンを含有するポリメタクリレート等の高分子材料を用いることができる。

【0027】正孔輸送層3aは、これらの正孔輸送材料を真空蒸着法、スパッタリング法、電子ビーム蒸着法等により成膜することにより、前記陽極2上に積層形成される。

【0028】真空蒸着法で正孔輸送層3aを形成する場合には、正孔輸送材料を真空容器内に設置されたルツボに入れ、真空容器内を適当な真空ポンプで 10^{-4} Pa程度にまで排気した後、ルツボを加熱して、正孔輸送材料を蒸発させ、ルツボに対向配置した基板1上の陽極2上に正孔輸送層3aを形成する。

【0029】このようにして正孔輸送層3aを形成する場合、更に、アクセプタとして、芳香族カルボン酸の金属錯体及び/又は金属塩、ベンゾフェノン誘導体及びチオベンゾフェノン誘導体、フラレン類等を 10^{-3} ~10重量%の濃度でドーピングして、フリーキャリアとしての正孔を生成させることにより、低電圧駆動を可能にすることができる。

【0030】正孔輸送層3aの膜厚は、通常、10~300nm、好ましくは30~100nmである。このような膜厚の薄い正孔輸送層を一樣に形成するためには、一般に真空蒸着法がよく用いられる。

【0031】また、正孔注入効率を更に向上させ、かつ、有機層全体の陽極2への付着力を改善する目的で、図4に示す如く、正孔輸送層3aと陽極2との間に正孔注入層3cを形成することも行われている。正孔注入層3cに用いられる材料としては、イオン化ポテンシャルが低く、導電性が高く、更に陽極2上で熱的に安定な薄膜を形成し得る材料が望ましく、フタロシアニン化合物やボルフィリン化合物が用いられる。このような正孔注入層3cを介在させることで、初期の素子の駆動電圧が下がると同時に、素子を定電流で連続駆動した時の電圧上昇も抑制される効果が得られる。正孔注入層3cもまた、正孔輸送層3aと同様にしてアクセプタをドーピングすることで導電性を向上させることが可能である。

【0032】正孔注入層3cの膜厚は、通常、2~100nm、好ましくは5~50nmである。このような膜厚の薄い正孔注入層を一樣に形成するためには、一般に真空蒸着法がよく用いられる。

【0033】正孔輸送層3aの上に形成される電子輸送層3bは、電界が与えられた電極間において、陰極からの電子を効率よく正孔輸送層3aの方向に輸送することができる化合物で構成される。

【0034】電子輸送層3bに用いられる電子輸送性化

化合物としては、陰極4からの電子注入効率が高く、かつ、注入された電子を効率よく輸送することができる化合物であることが必要である。そのためには、電子親和力が大きく、しかも電子移動度が大きく、更に安定性に優れ、製造時や使用時にトラップとなる不純物が発生しにくい化合物であることが要求される。

【0035】このような条件を満たす材料としては、テトラフェニルブタジエンなどの芳香族化合物、8-ヒドロキシキノリンのアルミニウム錯体などの金属錯体、シクロペンタジエン誘導体、ペリノン誘導体、オキサジアゾール誘導体、ビスチリルベンゼン誘導体、ペリレン誘導体、クマリン化合物、希土類錯体、ジスチリルピラジン誘導体、p-フェニレン化合物、チアジアゾロピリジン誘導体、ピロロピリジン誘導体、ナフチリジン誘導体などが挙げられる。

【0036】これらの化合物を用いた電子輸送層3bは、一般に、電子を輸送する役割と、正孔と電子の再結合の際に発光をもたらす役割とを同時に果たすことができる。

【0037】正孔輸送層3aが発光機能を有する場合、電子輸送層3bは電子を輸送する役割だけを果たす場合もある。

【0038】素子の発光効率を向上させるとともに発光色を変える目的で、例えば、8-ヒドロキシキノリンのアルミニウム錯体をホスト材料として、クマリン等のレーザ用蛍光色素をドーピングすること等も行われているが、本発明においても、上記の有機電子輸送性材料をホスト材料として各種の蛍光色素を 10^{-3} ~10モル%ドーピングすることにより、素子の発光特性をより一層向上させることができる。

【0039】電子輸送層3bの膜厚は、通常、10~200nm、好ましくは30~100nmである。

【0040】電子輸送層3bも正孔輸送層3aと同様の方法で形成することができるが、通常は真空蒸着法が用いられる。

【0041】なお、図2に示すような機能分離を行わない単層型の有機発光層3としては、先に挙げたポリ(p-フェニレンビニレン)、ポリ[2-メトキシ-5-(2-エチルヘキシルオキシ)-1,4-フェニレンビニレン]、ポリ(3-アルキルチオフェン)等の高分子材料や、ポリビニルカルバゾール等の高分子に発光材料と電子移動材料を混合した系等が挙げられる。

【0042】陰極4は、有機発光層3に電子を注入する役割を果たす。陰極4として用いられる材料は、前記陽極2に使用される材料を用いることが可能であるが、効率よく電子注入を行うには、仕事関数の低い金属が好ましく、スズ、マグネシウム、インジウム、カルシウム、アルミニウム、銀等の適当な金属又はそれらの合金が好適である。陰極4の膜厚は、通常、陽極2と同程度である。

【0043】低仕事関数金属からなる陰極を保護する目的で、この陰極上に更に、仕事関数が高く大気に対して安定な金属層を積層することにより、素子の安定性を増すことができる。この目的のための金属層には、アルミニウム、銀、ニッケル、クロム、金、白金等の金属が用いられる。

【0044】なお、図2~4は、本発明で採用される素子本体の一例を示すものであって、本発明は、図示のもの以外に、以下に示すような層構成の素子本体に適用することができる。

【0045】陽極/正孔輸送層/電子輸送層/界面層/陰極、陽極/正孔輸送層/電子輸送層/他の電子輸送層/陰極、陽極/正孔輸送層/電子輸送層/他の電子輸送層/界面層/陰極、陽極/正孔注入層/正孔輸送層/電子輸送層/界面層/陰極、陽極/正孔注入層/正孔輸送層/電子輸送層/他の電子輸送層/陰極

上記層構成で、界面層は陰極と有機層とのコンタクトを向上させるためのもので、芳香族ジアミン化合物、キナクリドン化合物、ナフタセン誘導体、有機シリコン化合物、有機リン化合物、N-フェニルカルバゾール骨格を有する化合物、N-ビニルカルバゾール重合体等で構成された層が例示できる。界面層の膜厚は、通常、2~100nm、好ましくは5~30nmである。界面層を設ける代わりに、有機発光層及び電子輸送層の陰極界面近傍に上記界面層の材料を50重量%以上含む領域を設けてもよい。

【0046】また、他の電子輸送層は、有機電界発光素子の発光効率を更に向上させるために、電子輸送層の上にさらに積層形成されるものであり、この電子輸送層に用いられる化合物には、陰極からの電子注入が容易で、電子の輸送能力が更に大きいことが要求される。このような電子輸送性材料としては、オキサジアゾール誘導体やそれらをポリメタクリル酸メチル(PMMA)等の樹脂に分散した系、フェナントロリン誘導体、又は、n型水素化非晶質炭化シリコン、n型硫化亜鉛、n型セレン化亜鉛等が挙げられる。この他の電子輸送層の膜厚は、通常、5~200nm、好ましくは10~100nmである。

【0047】このような有機電界発光素子の安定性及び信頼性を向上させるためには、素子全体、即ち、基板上に形成された陽極、有機発光層、陰極等の層状堆積物の全体をシールする必要がある。

【0048】以下に、本発明に係るシーリングを図5を参照して説明する。なお、図5には、図4に示した構造の有機電界発光素子本体10Bの陰極4の上に保護層5を設けてシーリングした場合を例示しているが、図2、3に示す有機電界発光素子の本体10、10A、或いはその他の層構成の有機電界発光素子本体であっても良いことは言うまでもない。

【0049】図5において、保護層5は、その下の陰極

4及び有機層を保護する目的で設けられており、 SiO_x 、 GeO 、 MgO 、 Ta_2O_5 などの酸化物、 SiN_x などの窒化物、 GeS などの硫化物、 MgF_2 などのフッ化物などが用いられる。その形成方法としては、抵抗加熱による真空蒸着法、スパッタ法、電子ビーム蒸着法などが用いられる。このうち、保護層形成時のダメージを抑制する目的で真空蒸着法が多く用いられるが、低ダメージ化したスパッタ法や、真空蒸着法とスパッタ法を併用することも行われている。保護層5の厚さは、一般に100nm～10 μm とされ、膜の緻密さや応力、その他の特性や要求性能に応じて適宜決定される。

【0050】保護層5を形成した後は、基板1の周辺など特定部分に引き出した電極部分を除いて、素子部(層状堆積物)を外界から遮断する目的でシーリングを行う。シーリング部材は少なくともシーリング剤(封止剤)6と背面ガラス7から構成されており、シーリング剤6を基板1或いは背面ガラス7に塗布したのち両者を接触させ、シーリング剤6の硬化を行ってシーリングを完了する。

【0051】本発明においては、このシーリングに当たり、保護層5を形成した後の基板1を大気に晒すことなくドライボックス中に採り、このシーリング作業を行う。

【0052】ここでいうドライボックスとは、通常の大気中に比べて環境ガス中に占める水分量(水蒸気量)が低くコントロールされた空間のことをいい、環境ガスとしては空気、窒素やアルゴンなどの不活性ガス、その他取り扱い物質と反応しないガスを用いることができる。ドライ環境にする方法としては、例えばアルゴンなどの乾燥したガスでボックス内を置換し、その後も少量ずつガスを流し続けることによりドライ環境を保つ方法がある。この方法では、露点約-10℃(約1600ppm)が可能である。更に低水分量にするには、環境ガスを循環精製させる装置を別途設置し、循環運転する方法があり、この方法によれば露点約-100℃(約0.06ppm)以下を維持することが可能である。

【0053】シーリング剤6としては、熱硬化性樹脂、光硬化性樹脂などが用いられる。いずれの樹脂においても、アクリル系、シリコン系に比べてエポキシ系樹脂が透湿性の点で優れており一般によく用いられる。シーリング剤6の厚みは、基板1に形成された素子(層状堆積物)の合計の厚みより厚い必要がある。このためシーリング部の厚みを確保する目的で、スペーサーとしてシリカビーズやガラスファイバー等がシーリング剤と一緒に用いられる場合がある。

【0054】また、背面の封止材としては、背面ガラス7の他に金属や樹脂などを用いても良いが、いずれの封止材においても、シーリング剤と同様に耐透湿性、耐酸素透過性などが要求される。封止材は、また、背面封止材の可視透過性、強度、可変性などの性質も考慮して、素子の用途に合わせて選択使用される。

【0055】次に、図1を参照して、このような有機電

界発光素子を製造するための本発明の有機電界発光素子の製造装置及びこの装置による有機電界発光素子の製造方法について説明する。

【0056】図1は本発明の有機電界発光素子の製造装置の実施の形態を示す模式図である。

【0057】図1中、21及び27はドライボックス、22～26は作業用真空室、22a～26aは搬送用真空室、22b～26bは基板及びマスク搬送用ロボットアーム、22c～27cはドライボックスと搬送用真空室又は搬送用真空室同士を連結するゲートバルブ、22d～26dは作業用真空室と搬送用真空室とを連結するゲートバルブである。

【0058】有機電界発光素子の製造に当たっては、基板1上にパターンニングされた陽極2が形成されたものをドライボックス21内に設置し、基板表面をUVオゾン洗浄した後、ドライボックス21内を不活性ガスで十分に置換する。次にゲートバルブ22cを開けてロボットアーム22bを用いて搬送用真空室22a内に基板を導入する。ゲートバルブ22cを閉じた後搬送用真空室22aを 10^{-5}Torr 以下に真空引きし、その後ゲートバルブ22dを開けてロボットアーム22bを用いて基板を移送し、作業用真空室22内に基板を設置する。作業用真空室22ではアルゴンと酸素の混合ガスで基板表面をプラズマ処理する。次に、真空を保持したままロボットアーム22bと23bを順次用いて、ゲートバルブ22d、搬送用真空室22a、ゲートバルブ23c、搬送用真空室23a、ゲートバルブ23dを通して基板を移送し、作業用真空室23内に基板を設置する。作業用真空室23内では正孔注入層3c及び正孔輸送層3aが順次蒸着形成され、その後ロボットアーム23b、24bを順次用いて、上記と同様に基板を移送して作業用真空室24内に基板を設置する。作業用真空室24内では電子輸送層3bが蒸着形成され、その後ロボットアーム24b、25bを順次用いて、上記と同様に基板を移送して作業用真空室25内に基板を設置する。作業用真空室25内では陰極4が蒸着形成され、その後ロボットアーム25b、26bを順次用いて、上記と同様に基板を移送し、作業用真空室26内に基板を設置する。作業用真空室26内では保護層5が蒸着形成され、その後ロボットアーム26bにより基板を搬送用真空室26a内に設置し、ゲートバルブ26dを閉じ搬送用真空室26a内に不活性ガスを大気圧まで充填する。

【0059】次に、ゲートバルブ27cを開け、予め不活性ガスで満たされたドライボックス27内にロボットアーム26bを用いて基板を設置する。ドライボックス27内では、シーリング剤6の光硬化性樹脂を基板1上に塗布した後、背面ガラス7に接触させて有機電界発光素子本体10Bをシールする。

【0060】以上のように、この装置によれば、ドライボックス21内でUVオゾン洗浄後不活性ガスで充填さ

れてからドライボックス27でシールが完了するまで、基板は大気に晒されることはない。

【0061】このようにしてシールが完了した有機電界発光素子は、ドライボックス27から取り出され、素子駆動用の電気配線や半導体回路の実装がなされ、パネルとして完成される。

【0062】なお、図1では、作業用真空室及び搬送用真空室等で構成される移送及び処理用ユニットが5個直列に直線状に連結されているが、この連結ユニット数は必ずしも5個である必要はなく、層状堆積物数や層状堆積物の種類、その他基板の処理方法等により2以上の任意の数を連結でき、また、層状堆積物の増減や処理方法の変更等に応じて、ユニットの数を増減することも容易に行える。また、ドライボックスも必ずしも2個必要ではなく、2つの機能を1つに集約したり、一方の機能を省略することも可能である。ロボットアームは搬送用真空室に1つ以上あることが必要で、これを複数台設けた場合には、基板やマスクの移送が効率的になり、作業時間をより短縮できる。

【0063】ここでいうロボットアームとは、真空空間を基板やマスクを保持して移動できる機構のことを指し、装置の形状、基板の保持手段或いは移動手段を何ら規定するものではなく、例えば、コンベア式移動手段などとの組み合わせも可能である。

【0064】また、作業用真空室22～26における処理は、上記に説明した方法に何ら限定されず、成膜方法としては、スパッタ法、電子ビーム法など様々な処理方法を採用可能である。また、作業用真空室22における基板の処理方法としてもアルゴンガスと酸素ガスの混合ガスによるプラズマ処理法以外に、紫外線やエキシマレーザーを照射したり、特定のガスに晒して基板表面を改質する方法など様々な基板処理方法を採用できる。例えば、作業用真空室22においてプラズマ又はレーザー照射により基板の改質を行い、作業用真空室23～25において抵抗加熱による蒸着法で各有機層及び陰極を形成し、作業用真空室26においてスパッタ法により保護層を形成することもできる。また、成膜方法及び基板処理方法は、1つの作業用真空室において複数の方法を持つこともでき、それぞれの方法を用いて順次、或いは同時に成膜或いは処理することも可能である。また、ここでは下部電極として陽極をパターン形成した後の処理方法を記載したが、下部電極も図示の装置において形成することも可能である。また、陽極及び陰極の形成順も固定されたものではなく、有機層の構成に合わせて自由に選択できる。

【0065】本発明は、単一の素子、アレイ状に配置された構造からなる素子、陽極と陰極がX-Yマトリックス状に配置された構造のいずれの有機電界発光素子に対しても適用可能である。

【0066】

【実施例】次に、実施例及び比較例を挙げて本発明を更に具体的に説明するが、本発明はその要旨を超えない限り、以下の実施例の記載に限定されるものではない。

【0067】実施例1

ガラス基板として厚さ1.1mmのコーニング社製7059ガラスを用い、その上にインジウム・スズ酸化物(ITO)透明導電膜を120nm堆積(ジオマテック社製電子ビーム成膜品;シート抵抗20Ω)し、ITO膜付きガラス基板を得た。

【0068】次に、図1に示す装置を用いて図5に示す構造を有する有機電界発光素子を以下の方法で作製した。

【0069】ガラス基板1上に堆積されたITO透明導電膜を通常のフォトリソグラフィ技術と塩酸エッチングを用いて2mm幅のストライプにパターンニングして陽極2を形成した。パターン形成したITO基板を、アセトンによる超音波洗浄、純水による水洗、イソプロピルアルコールによる超音波洗浄の順で洗浄後、窒素ブローで乾燥させ、ドライボックス21内に設置した。ドライボックス21内ではUV/オゾン洗浄を10分間行った後、ドライボックス内を窒素ガスでガス置換し、ゲートバルブ22cを開けてロボットアーム22bにより搬送用真空室22a内に基板を導入した。ゲートバルブ22cを閉じた後、クライオポンプをも用いて搬送用真空室22a内を 1.1×10^{-6} Torr (約 1.5×10^{-4} Pa)まで真空引きした。その後、ゲートバルブ22dを開けロボットアーム22bを用いて作業用真空室22内に基板1を設置した。ゲートバルブ22dを閉じた後、 1.0×10^{-6} Torrの真空状態であった作業用真空室22内にアルゴン50%、酸素50%の混合ガスを 1.0×10^{-3} Torrになるまで導入し、プラズマ中でITO付きガラス基板1の表面を5分間処理した。その後ガスの導入を停止し、再び 1.0×10^{-6} Torrの真空状態になるまで作業用真空室22内を真空引きした。

【0070】次に、ゲートバルブ22dを開けロボットアーム22bを用いて基板1を作業用真空室22から搬送用真空室22a内に移送し、ゲートバルブ22dを閉じた後ゲートバルブ23cを開けて、ロボットアーム22b及びロボットアーム23bを用いて基板1を搬送用真空室23a内に移送し、ゲートバルブ23cを閉じた。基板1が搬送用真空室22aから23aに移送される間、搬送用真空室22a、23a内は 1.0×10^{-6} Torrの真空状態に保たれていた。

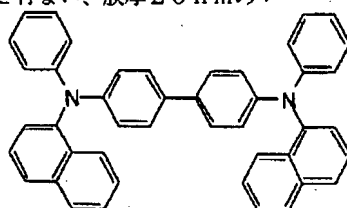
【0071】次に、ゲートバルブ23dを開けて、ロボットアーム23bを用いて作業用真空室23内に基板1を設置した。この際、基板1は予め配置されていた金属マスクの上に設置され、基板1上の陽極取り出し用の部分は金属マスクによりカバーされるようにした。次に、ゲートバルブ23dを閉じ、作業用真空室23内に配置

されたモリブデンボートに入れた以下に示す銅フタロシ
アニン (H1) (結晶形はβ型) を加熱して蒸着を行っ
た。真空度 1.1×10^{-6} Torr (約 1.5×10^{-4}
Pa)、蒸着時間1分で蒸着を行ない、膜厚20nmの*

*正孔注入層3cを得た。

【0072】

【化1】

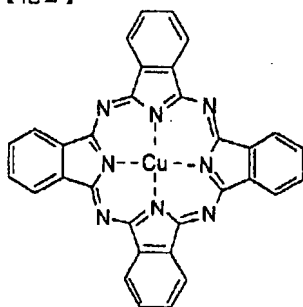


(H1)

【0073】次に、作業用真空室23内に配置されたセ
ラミックスるつぽに入れた、以下に示す、4,4'-ビ
ス[N-(1-ナフチル)-N-フェニルアミノ]ビフ
ェニル (H2) をるつぽの周囲のタンタル線ヒーターで
加熱して正孔注入層3cの上に積層した。この時のるつ
ぽの温度は、230~240℃の範囲で制御した。蒸着
時の真空度 8×10^{-7} Torr (約 1.1×10^{-4} P
a)、蒸着時間1分50秒で膜厚60nmの正孔輸送層
3aを得た。

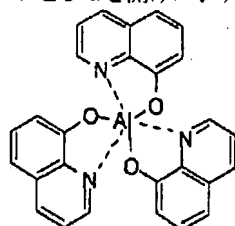
【0074】

【化2】



(H2)

【0075】次に、ゲートバルブ23dを開けロボット※



(E1)

【0077】なお、上記の正孔注入層3c、正孔輸送層
3a及び電子輸送層3bを真空蒸着する時の基板温度は
室温に保持した。

【0078】次に、ゲートバルブ24dを開けロボット
アーム24bを用いて基板1を作業用真空室24から搬
送用真空室24a内に移送し、ゲートバルブ24dを閉
じた後ゲートバルブ25cを開けて、ロボットアーム2
4b及びロボットアーム25bを用いて基板1を搬送用
真空室25a内に移送し、ゲートバルブ25cを閉じ
た。その後ゲートバルブ25dを開けて、ロボットア
ーム25bを用いて作業用真空室25内に基板1を設置し★50

※アーム23bを用いて基板1を作業用真空室23から搬
送用真空室23a内に移送し、ゲートバルブ23dを閉
じた後ゲートバルブ24cを開けて、ロボットアーム2
3b及びロボットアーム24bを用いて基板1を搬送用
真空室24a内に移送し、ゲートバルブ24cを閉じ
た。その後、ゲートバルブ24dを開けて、ロボットア
ーム24bを用いて作業用真空室24内に基板1を設置
した。この際、基板1は予め配置されていた金属マスク
の上に設置され、基板1上の陽極取り出し用の部分は金
属マスクによりカバーされるようにした。次にゲートバ
ルブ24dを閉じ、作業用真空室24内にて、発光機能
を有する電子輸送層3bの材料として、以下の構造式で
示すアルミニウムの8-ヒドロキシキノリン錯体A1
(C_9H_6NO)₃ (E1) を上記正孔輸送層3aの上
に同様に蒸着を行った。この時のるつぽの温度は3
10~320℃の範囲で制御した。蒸着時の真空度は 9
 $\times 10^{-7}$ Torr (約 1.2×10^{-4} Pa)、蒸着時間
は2分40秒で、蒸着された電子輸送層3bの膜厚は7
5nmであった。

30 【0076】

【化3】

40★た。この際、基板1は予め配置されていた陰極蒸着用の
金属マスクの上に設置され、2mm幅のストライプ状シ
ャドーマスクを、パターン化された陽極2のITOスト
ライプとは直交するように基板1に密着させてた。

【0079】次に、ゲートバルブ25dを閉じ、作業用
真空室25内にて、陰極4として、マグネシウムと銀の
合金電極を2元同時蒸着法によって膜厚100nmとな
るように蒸着した。蒸着はモリブデンボートを用いて、
真空度 1×10^{-5} Torr (約 1.3×10^{-3} Pa)、
蒸着時間3分10秒で行った。また、マグネシウムと銀
の原子比は10:1.2とした。更に続いて、作業用真

空室25内において、モリブデンボートを用いてアルミニウムを100nmの膜厚でマグネシウム・銀合金膜の上に積層して陰極4を完成させた。アルミニウム蒸着時の真空度は 2.3×10^{-5} Torr (約 3.1×10^{-3} Pa)、蒸着時間は1分40秒であった。以上のマグネシウム・銀合金とアルミニウムの2層型陰極の蒸着時の基板温度は室温に保持した。

【0080】次に、ゲートバルブ25dを開けロボットアーム25bを用いて基板1を作業用真空室25から搬送用真空室25a内に移送し、ゲートバルブ25dを閉じた後ゲートバルブ26cを開けて、ロボットアーム25b及びロボットアーム26bを用いて基板1を搬送用真空室26a内に移送し、ゲートバルブ26cを閉じた。その後ゲートバルブ26dを開けて、ロボットアーム26bを用いて作業用真空室26内に基板1を設置した。この際、基板1を予め配置されていた保護層蒸着用の金属マスクの上に密着して設置した。次に、ゲートバルブ26dを閉じ、作業用真空室26内にて、保護層5として、酸化ケイ素(SiO_x ; $x=1.0 \sim 2.0$)を膜厚1000nmとなるように蒸着した。蒸着はモリブデンボートを用いて、真空度 1×10^{-5} Torr (約 1.3×10^{-3} Pa)、蒸着時間3分40秒で行った。この蒸着時の基板温度は室温に保持した。これにより、陽極及び陰極の取り出し部分を除いた素子部は保護層5によりカバーされた。

【0081】次に、ゲートバルブ26dを開けロボットアーム26bを用いて基板1を作業用真空室26から搬送用真空室26a内に移送し、ゲートバルブ26dを閉じた後、搬送用真空室26a内に窒素を導入し大気圧とした。

【0082】その後、ゲートバルブ27cを開けて、予め窒素ガスで満たされていたドライボックス27内にロボットアーム26bを用いて基板1を設置した。次に、シール剤6としてスリーボンド社製の光硬化性樹脂(30Y-184)を、有機電界発光素子本体10Bの基板1上のシール部に、幅0.5mmでデイス Pensした。その後、予めシール部のサイズにカットされた背面ガラス7を、シール剤6が塗付された部分に設置し、ガラス基板1と背面ガラス6が密着するように50g/cm²の圧力を30秒間かけた。

【0083】次に、シール剤6が塗付された部分に紫外光(波長365nm)を4.2J/cm²照射し、光硬化性樹脂を硬化させ、シールを完了させた。硬化の際には、紫外線及び熱による素子の劣化を防ぐためにシール部以外の部分は遮光した。

【0084】その後、ドライボックス27に設けられた取り出し室から素子を取り出し、2mm×2mmのサイズの有機電界発光素子を得た。

【0085】得られた有機電界発光素子の陽極2にプラス、陰極4にマイナスの直流電圧を印加して発光させ、

発光特性を測定した。電流密度15mA/cm²の電流を素子に流し続けたときの発光輝度の初期値に対する時間変化を図6に示す。

【0086】本実施例による有機電界発光素子の通電初期の発光輝度は9250cd/m²であった。10000時間経過後の発光輝度は8650cd/m²となり、初期値に対する相対輝度は0.935であり輝度の低下は小さかった。

【0087】比較例1

10 比較のために、実施例1で製造した有機電界発光素子と同じ層構成の有機電界発光素子を別の方法で作製した。

【0088】即ち、複数の作業用真空室が連結することなく独立した装置を用いて、電子輸送層3bの形成後と保護層5の形成後の2回にわたり、基板1を作業用真空室から大気中に一旦出して次の工程に移したことを除き、他は実施例1と同様の条件で有機電界発光素子を作製した。

【0089】得られた有機電界発光素子は、層の構成、材質、膜厚及び外観は実施例1のものと同一であった。

20 【0090】この有機電界発光素子について、実施例1と同様の方法で発光輝度の測定を行い、発光輝度の初期値に対する時間変化を図6に示した。

【0091】本比較例の有機電界発光素子は、通電初期の発光輝度は9100cd/m²であり、実施例1のものと大きな差はなかった。しかしながら、10000時間経過後の発光輝度は5105cd/m²で、初期値に対する相対輝度は0.561となり、また、10000時間後には発光輝度の測定が不可能なほど光らなくなった。

30 【0092】比較例2

比較のために、実施例1で製造した有機電界発光素子と同じ層構成の有機電界発光素子を更に別の方法で作製した。

【0093】即ち、1つの搬送用真空室の周囲に複数の作業用真空室が連結された装置を用いて、保護層5の形成後に基板1を作業用真空室から大気中に一旦出して次のシール工程に移したことを除き、他は実施例1と同様の条件で有機電界発光素子を作製した。

40 【0094】得られた有機電界発光素子は、層の構成、材質、膜厚及び外観は実施例1のものと同一であった。

【0095】有機電界発光素子について、実施例1と同様の方法で発光輝度の測定を行い、発光輝度の初期値に対する時間変化を図6に示した。

【0096】本比較例の有機電界発光素子は、通電初期の発光輝度は9150cd/m²であり、実施例1のものと大きな差はなかった。しかしながら、10000時間経過後の発光輝度は6680cd/m²で、初期値に対する相対輝度は0.73となり比較例1よりは輝度の低下が小さかったものの、実施例1に比べると輝度の低下が大きかった。

【0097】

【発明の効果】以上詳述した通り、本発明によれば、有機電界発光素子の製造に当たり、陽極のパターン形成後からシーリングまでの各工程及びその移行工程も含めて一貫して基板を大気に晒すことなく実施することで、各層状堆積物が大気中の水分や酸素に接触するのを防止して、発光特性及び発光寿命特性に優れた有機電界発光素子を生産性良く、かつ低コストで提供することができる。

【0098】本発明で製造される有機電界発光素子は、フラットパネル・ディスプレイ（例えばOAコンピュータ用や壁掛けテレビ）や面発光体としての特徴を生かした光源（例えば、複写機の光源、演奏ディスプレイや計器類のバックライト光源）、表示板、標識灯への応用が期待され、その技術的価値は極めて大きい。

【図面の簡単な説明】

【図1】本発明の有機電界発光素子の製造装置の実施の形態を示す模式的断面図である。

【図2】本発明で製造される有機電界発光素子本体の実施例を示す模式的断面図である。

【図3】本発明で製造される有機電界発光素子本体の他の実施例を示す模式的断面図である。

【図4】本発明で製造される有機電界発光素子本体の別の実施例を示す模式的断面図である。

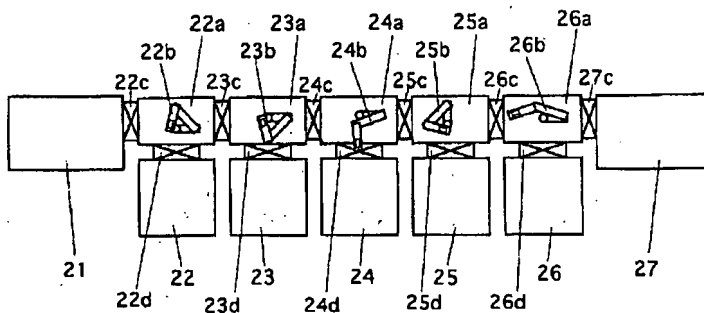
【図5】本発明で製造される有機電界発光素子の実施例を示す模式的断面図である。

【図6】実施例1及び比較例1、2における有機電界発光素子の初期発光輝度に対する相対発光輝度の駆動発光時間依存性を示すグラフである。

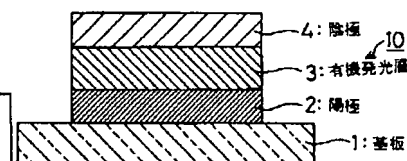
【符号の説明】

- 1 基板
- 2 陽極
- 3 有機発光層
- 3a 正孔輸送層
- 3b 電子輸送層
- 3c 正孔注入層
- 4 陰極
- 5 保護層
- 6 シール剤
- 7 背面ガラス
- 10, 10A, 10B 有機電界発光素子本体
- 21, 27 ドライボックス
- 22, 23, 24, 25, 26 作業用真空室
- 22a, 23a, 24a, 25a, 26a 搬送用真空室
- 22b, 23b, 24b, 25b, 26b ロボットアーム
- 22c, 23c, 24c, 25c, 26c, 27c ゲートバルブ
- 22d, 23d, 24d, 25d, 26d ゲートバルブ

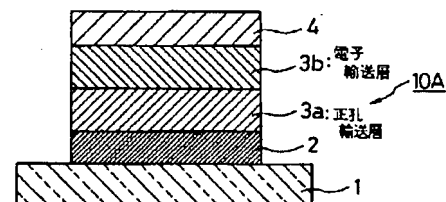
【図1】



【図2】



【図3】



22b-26b